

WHAT IS CLAIMED IS:

1. A process for making a blocked mercaptosilane, comprising the steps of:

reacting mercaptosilane with organic or inorganic halide or anhydride in the presence of at least one acid acceptor to produce a slurry reaction product, wherein said acid acceptor in free form is substantially water insoluble and the respective salt of said acid acceptor is substantially water soluble;

washing the slurry reaction product with water or brine to produce an organic phase that comprises the blocked mercaptosilane and an aqueous phase that is immiscible with the organic phase and that comprises the respective salt of the acid acceptor.

2. The process of claim 1 wherein the acid acceptor is a tertiary amine having an aqueous solubility less than 0.2% and the respective salt is a tertiary amine hydrohalide.

3. The process of claim 1 wherein the mercaptosilane is at least one of those providing the respective blocked mercaptosilane selected from the group of 2-triethoxysilyl-1-ethyl thioacetate; 2-trimethoxysilyl-1-ethyl thioacetate; 2-(methyldimethoxysilyl)-1-ethyl thioacetate; 3-trimethoxysilyl-1-propyl thioacetate; triethoxysilylmethyl thioacetate; trimethoxysilylmethyl thioacetate; triisopropoxysilylmethyl thioacetate; methyldiethoxysilylmethyl thioacetate; methyldimethoxysilylmethyl thioacetate; methyldiisopropoxysilylmethyl thioacetate; dimethylethoxysilylmethyl thioacetate; dimethylmethoxysilylmethyl thioacetate; dimethylisopropoxysilylmethyl thioacetate; 2-triisopropoxysilyl-1-ethyl thioacetate; 2-(methyldiethoxysilyl)-1-ethyl thioacetate; 2-(methyldiisopropoxysilyl)-1-ethyl thioacetate; 2-(dimethylethoxysilyl)-1-ethyl thioacetate; 2-(dimethylmethoxysilyl)-1-ethyl thioacetate; 2-(dimethylisopropoxysilyl)-1-ethyl thioacetate; 3-triethoxysilyl-1-propyl thioacetate; 3-triisopropoxysilyl-1-propyl thioacetate; 3-methyldiethoxysilyl-1-propyl thioacetate; 3-methyldimethoxysilyl-1-propyl thioacetate; 3-methyldiisopropoxysilyl-1-propyl thioacetate; 1-(2-triethoxysilyl-1-ethyl)-4-

thioacetylcyclohexane; 1-(2-triethoxysilyl-1-ethyl)-3-thioacetylcyclohexane;
 2-triethoxysilyl-5-thioacetylnorbornene; 2-triethoxysilyl-4-thioacetylnorbornene;
 2-(2-triethoxysilyl-1-ethyl)-5-thioacetylnorbornene; 2-(2-triethoxysilyl-1-ethyl)-4-
 thioacetylnorbornene; 1-(1-oxo-2-thia-5-triethoxysilylpenyl)benzoic acid;
 6-triethoxysilyl-1-hexyl thioacetate; 1-triethoxysilyl-5-hexyl thioacetate; 8-
 triethoxysilyl-1-octyl thioacetate; 1-triethoxysilyl-7-octyl thioacetate; 6-triethoxysilyl-
 1-hexyl thioacetate; 1-triethoxysilyl-5-octyl thioacetate; 8-trimethoxysilyl-1-octyl
 thioacetate; 1-trimethoxysilyl-7-octyl thioacetate; 10-triethoxysilyl-1-decyl thioacetate;
 1-triethoxysilyl-9-decyl thioacetate; 1-triethoxysilyl-2-butyl thioacetate; 1-
 triethoxysilyl-3-butyl thioacetate; 1-triethoxysilyl-3-methyl-2-butyl thioacetate; 1-
 triethoxysilyl-3-methyl-3-butyl thioacetate; 3-trimethoxysilyl-1-propyl thiooctanoate;
 3-triethoxysilyl-1-propyl thiopalmitate; 3-triethoxysilyl-1-propyl thiooctanoate; 3-
 triethoxysilyl-1-propyl thiobenzoate; 3-triethoxysilyl-1-propyl thio-2-ethylhexanoate;
 3-methyldiacetoxysilyl-1-propyl thioacetate; 3-triacetoxysilyl-1-propyl thioacetate; 2-
 methyldiacetoxysilyl-1-ethyl thioacetate; 2-triacetoxysilyl-1-ethyl thioacetate; 1-
 methyldiacetoxysilyl-1-ethyl thioacetate; 1-triacetoxysilyl-1-ethyl thioacetate; tris-(3-
 triethoxysilyl-1-propyl)trithiophosphate; bis-(3-triethoxysilyl-1-
 propyl)methyldithiophosphonate; bis-(3-triethoxysilyl-1-
 propyl)ethyldithiophosphonate; 3-triethoxysilyl-1-propyldimethylthiophosphinate; 3-
 triethoxysilyl-1-propyldiethylthiophosphinate; tris-(3-triethoxysilyl-1-
 propyl)tetrathiophosphate; bis-(3-triethoxysilyl-1-propyl)methyltrithiophosphonate;
 bis-(3-triethoxysilyl-1-propyl)ethyltrithiophosphonate; 3-triethoxysilyl-1-
 propyldimethyldithiophosphinate; 3-triethoxysilyl-1-propyldiethyldithiophosphinate;
 tris-(3-methyldimethoxysilyl-1-propyl)trithiophosphate; bis-(3-methyldimethoxysilyl-
 1-propyl)methyldithiophosphonate; bis-(3-methyldimethoxysilyl-1-
 propyl)ethyldithiophosphonate; 3-methyldimethoxysilyl-1-
 propyldimethylthiophosphinate; 3-methyldimethoxysilyl-1-
 propyldiethylthiophosphinate; 3-triethoxysilyl-1-propylmethylthiosulphate; 3-
 triethoxysilyl-1-propylmethanethiosulphonate; 3-triethoxysilyl-1-
 propylethanethiosulphonate; 3-triethoxysilyl-1-propylbenzenethiosulphonate; 3-
 triethoxysilyl-1-propyltoluenethiosulphonate; 3-triethoxysilyl-1-

propylnaphthalenethiosulphonate; 3-triethoxysilyl-1-propylxylenethiosulphonate; triethoxysilylmethylmethylthiosulphate; triethoxysilylmethylmethanethiosulphonate; triethoxysilylmethylethanethiosulphonate; triethoxysilylmethylbenzenethiosulphonate; triethoxysilylmethyltoluenethiosulphonate; triethoxysilylmethylnaphthalenethiosulphonate; and triethoxysilylmethylxylenethiosulphonate.

4. The process of claim 1, wherein the organic or inorganic halide or anhydride is at least one of those providing the respective blocked mercaptosilanes selected from the group of compounds listed in claim 3.

5. The process of claim 1 wherein the organic phase further comprises an inert solvent, a portion of which is recycled from a previous run.

6. The process of claim 1 wherein the slurry reaction product is formed in the temperature range of 0-150° C under an inert atmosphere.

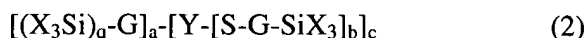
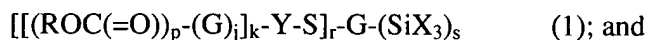
7. The process of claim 1 wherein the steps are performed continuously with neutralization of said respective salt of said acid acceptor and recycle of said acid acceptor.

8. The process of claim 1 wherein the mercaptosilane is selected from 3-mercaptopropyltriethoxysilane, 3-mercaptopropylmethyldiethoxysilane, 3-mercaptopropyldimethylethoxysilane, 3-mercaptopropyltrimethoxysilane, 3-mercaptopropylmethyldimethoxysilane, and 3-mercaptopropyldimethylmethoxysilane, and the organic halide is selected from octanoyl chloride, 2-ethylhexanoyl chloride, hexanoyl chloride, decanoyl chloride, lauroyl chloride, and oleoyl chloride, and the acid acceptor is tripropylamine.

9. The process of claim 1 wherein the blocked mercaptosilane is 3-triethoxysilyl-1-propyl thiooctanoate, and the molar ratios of mercaptosilane to tripropylamine to acid chloride range from 1.00 / 1.05 / 1.00 to 1.25 / 5.00 / 1.00.

10. The process of claim 5 wherein the organic solvent is selected from the group of hydrocarbons including alkanes, isoalkanes, petroleum ethers, cycloalkanes, and aromatics.

11. The process of claim 1 wherein the mercaptosilane has the formula:



wherein

Y is a polyvalent species $(\text{Q})_z\text{A}(=\text{E})$ selected from the group consisting of:

-C(=NR)-; -SC(=NR)-; -SC(=O)-; (-NR)C(=O)-; (-NR)C(=S)-; -OC(=O)-; -OC(=S)-; -C(=O)-; -SC(=S)-; -C(=S)-; -S(=O)-; -S(=O)₂-; -OS(=O)₂-; (-NR)S(=O)₂-; -SS(=O)-; -OS(=O)-; (-NR)S(=O)-; -SS(=O)₂-; (-S)₂P(=O)-; (-S)P(=O)-; -P(=O)(-)₂; (-S)₂P(=S)-; (-S)P(=S)-; -P(=S)(-)₂; (-NR)₂P(=O)-; (-NR)(-S)P(=O)-; (-O)(-NR)P(=O)-; (-O)(-S)P(=O)-; (-O)₂P(=O)-; (-O)P(=O)-; (-NR)P(=O)-; (-NR)₂P(=S)-; (-NR)(-S)P(=S)-; (-O)(-NR)P(=S)-; (-O)(-S)P(=S)-; (-O)₂P(=S)-; (-O)P(=S)-; and -(-NR)P(=S)-; each wherein the atom (A) attached to the unsaturated heteroatom (E) is attached to the sulfur, which in turn is linked via a group G to the silicon atom;

each R is chosen independently from hydrogen, straight, cyclic, or branched alkyl that may or may not contain unsaturation, alkenyl groups, aryl groups, and aralkyl groups, with each R containing from 1 to 18 carbon atoms;

each G is independently a monovalent or polyvalent group derived by substitution of alkyl, alkenyl, aryl, or aralkyl wherein G can contain from 1 to 18 carbon atoms, and if G directly bonded to Y is univalent (i.e., if p = 0), G can be a hydrogen atom;

X is independently selected from the group consisting of -Cl, -Br, RO-, RC(=O)O-, R₂C=NO-, R₂NO- or R₂N-, -R, -(OSiR₂)_i(OSiR₃) wherein each R and G is as above and at least one X is not -R;

Q is oxygen, sulfur, or (-NR-);

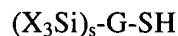
A is carbon, sulfur, phosphorus, or sulfonyl;

E is oxygen, sulfur, or NR;

p is 0 to 5; r is 1 to 3; z is 0 to 2; q is 0 to 6; a is 0 to 7; b is 1 to 3; j is 0 to 1, but it may be 0 only if p is 1; c is 1 to 6, preferably 1 to 4; t is 0 to 5; s is 1 to 3; k is 1 to 2; with the provisos that (A) if A is carbon, sulfur, or sulfonyl, then (i) $a + b = 2$ and (ii) $k = 1$; (B) if A is phosphorus, then $a + b = 3$ unless both (i) $c > 1$ and (ii) $b = 1$, in which case $a = c + 1$; and (C) if A is phosphorus, then k is 2.

and the organic halide is selected from octanoyl chloride, 2-ethylhexanoyl chloride, hexanoyl chloride, decanoyl chloride, lauroyl chloride, and oleoyl chloride, and the acid acceptor is tripropylamine.

12. The process of claim 1 wherein the mercaptosilane has the formula:



wherein X is independently selected from the group consisting of -Cl, -Br, RO-, $RC(=O)O-$, $R_2C=NO-$, R_2NO- or R_2N- , -R, $-(OSiR_2)_t(OSiR_3)$ wherein each R is chosen independently from hydrogen, straight, cyclic, or branched alkyl that may or may not contain unsaturation, alkenyl groups, aryl groups, and aralkyl groups, with each R containing from 1 to 18 carbon atoms, and at least one X is not -R; G is a monovalent or polyvalent group derived by substitution of alkyl, alkenyl, aryl, or aralkyl wherein G can contain from 1 to 18 carbon atoms;

and the organic halide is selected from octanoyl chloride, 2-ethylhexanoyl chloride, hexanoyl chloride, decanoyl chloride, lauroyl chloride, and oleoyl chloride, and the acid acceptor is tripropylamine.